PATENT SPECIFICATION

767,958

Date of Application and filing Complete Specification: Nov. 20, 1953. No. 32183/53.

Application made in Germany on Nov. 21, 1952.

Complete Specification Published: Feb. 13, 1957.

Index at acceptance:—Classes 70, Q5X; and 95, A5.

International Classification:—C08c, C09d.

COMPLETE SPECIFICATION

A Process for Improving the Surface Properties of Powders

We, DEHYDAG DEUTSCHE HYDRIERWERKE G.m.b.H., formerly known as DEHYDAG DEUTSCHE HYDRIERWERKE A.G., a German Company, of 67, Henkelstrasse, Dusseldorf, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the surface properties of powders.

It has been found that the surface properties of powders especially of substances which are employed in technology in the most varying fields of application under the heading of "pigments", can be considerably improved if these substances are impregnated with a mixture of a metal salt of a sulphonic acid or of an acid sulphuric acid ester and a salt of a sulphonic acid or of an acid sulphuric acid ester with ammonia or an organic base. The metal salt may be an alkali-, alkaline earth-, or earth-metal salt of an alkyl sulphuric acid, alkyl sulphonic acid or alkylbenzene sulphonic acid while the ammonium or organic base salt may be a salt of an alkyl sulphuric acid alkyl sulphonic acid or alkylbenzene sulphonic acid.

The ability of the powders to be moistened and their ability to be distributed in liquid, highly viscous or plastic organic materials is considerably improved by the impregnation, as are their floating capacity in the case of suspension in liquids, their power of adhesion and their ability to be poured.

Pigments thus impregnated possess especial practical interest, for example, for lacquers, lacquer colours, oil colours, synthetic resin lacquer colours, and industrial coatings, in which they show an excellent suspension capacity or as fillers for natural and synthetic rubber, rubber regenerates, and rubber substitutes.

The powders can be of an inorganic or an organic nature, and include inorganic oxides, hydroxides and salts such as are used, for instance, as colour pigments; for example ochre, red iron oxide, red lead, titanium dioxide, zinc sulphide, barium sulphate, and calcium carbonate; and further, carbon black, zinc oxide, finely dispersed dehydrated silicic acid, silica gel, calcium silicate dispersions or aluminium silicate dispersions, such as come into consideration as fillers for rubber and rubber regenerates. Further, mention should be made of powder bases, such as talcum, rice flour and starch, Fuller's earth, kaolin, pumice stone powder and alumina; and also active carbon, cork meal, wood meal and other known inorganic or organic powdery substances.

The impregnating of these powders with the mixtures of the aforementioned salts may be effected in the usual manner by stirring the powders into solutions of the salt mixtures in water or an organic solvent, filtering off the solution-impregnated powders and then drying them, or alternatively by sprinkling or spraying the solutions onto the dry pigments in a suitable manner. In appropriate cases, the salt mixtures can also be applied directly to the powders by grinding or crushing (in an edge mill), it being possible to employ the two components either dry or in a moistened condition. The quantities in which the salts can be applied to the powders depend on the working conditions applied and the particular purpose of use. These quantities lie between 0.05%-10% or more, and preferably from 0.1%-5%, calculated in relation to the powders.

The acid components of the salts included within the compass of the present invention include acid sulphuric acid esters of aliphatic, cycloaliphatic, and araliphatic alcohols and sulphonic acids with an aliphatically or aromatically bound sulphonic acid group, of which the following examples may be given:- hexylsulphuric acid, octylsulphuric acid, dodecylsulphuric acid, oleylsulphuric

[Price 3/-]

acid or mixtures of alkylsulphuric acids, acid sulphuric acid esters of naphthene alcohols or resin alcohols, hexylsulphonic acid, cyclohexylsulphonic acid, octylsulphonic acid, and alkylsulphonic acid mixtures, such as are obtained by the action of sulphur dioxide and chlorine upon hydrocarbon mixtures such as Diesel oil; further, octylbenzenesulphonic acid, dodecylbenzene sulphonic acid or other alkylbenzenesulphonic acids or their mixtures, such as are obtained by condensation of unsaturated benzine hydrocarbons with benzene in the presence of aluminium chloride followed by sulphonation, and furthermore tetrahydronaphthalenesulphonic acid.

The alkyl residues of these sulphuric acid derivatives can also contain heteroatoms, such as oxygen, sulphur, or nitrogen or atom groups deriving therefrom and also substituents.

The inorganic salts which are used for forming the salt mixtures according to the invention are, for example, sodium, potassium, calcium, magnesium and aluminium salts. Primary, secondary, or tertiary bases, such as octylamine, dioctylamine, cyclohexylamine, diethylcyclohexylamine, monomethylaniline, diethylaniline, monoethanolamine, triethanolamine, morpholine, piperidine, pyridine, and quinoline can be used as organic bases.

The impregnated powders or pigments are suitable for the most varied technical fields of application. Apart from the manufacture, already mentioned, of pigmentated lacquers and industrial coatings and the use in the rubber industry, they can, for example, be used in the mixing of powdered turbidifying, colouring or filling agents into synthetic substances or plastic masses, in the manufacture of powders for dusting rubber or synthetic resin sheets, and further in the manufacture of pigmented coatings for paper, and paper board or also as fillers for paper and paper boards, as a delustering agent for rayon and moulded threads, and as a filling, weighting or finishing agent in the textile industry.

The impregnating process according to the invention can consequently be employed in all cases in which it is desired to improve the surface properties of powders, increasing their strength in use, improving the stability of their state of distribution, and improving the useful properties of the products manufactured by the process.

EXAMPLE 1

500 parts by weight of red oxide are treated in a ball mill with 5 parts by weight of a mixture of 20% of the diethylcyclohexylamine salts of alkyl sulphuric acid mixture with alkyl residues C_3-C_{11} and 80% of the magnesium salts of an alkyl sulphuric acid mixture with alkyl residues C_{10}-C_{17} until a uniform distribution of the mixture on the red iron oxide has been achieved. The red iron oxide shows no alteration of its colour character, is stable to storage, and can be worked into lacquers and industrial coatings easily and homogeneously.

The pigment can, for example, be rubbed into a resin ester-linseed oil lacquer (1 : 1) in the ratio of 50 parts of pigment to 50 parts of lacquer on a cone mill. After an observation period of 6 months, the rubbing shows no sediment, but at the most, a concentration of pigment below the top third of the contents of the vessel, which can very easily be brought into uniform distribution again. The sediment formation, measured by the Boller-Lichthardt process (Farbe und Lack, Year 58, pp. 441-443), was so slight that the test cone reached the bottom of the vessel at a loading of only 9 gm. A rubbing of the same binding agent with the same but untreated, red iron oxide in the same quantity ratio produced, after an observation period of 6 months, a thick, tough sediment which could only be stirred up again with difficulty. By the above-mentioned method of testing, this rubbing required a cone loading of 150 gm. in order to reach the bottom of the vessel.

EXAMPLE 2

500 parts by weight of red iron oxide are treated in an edge mill with 5 parts by weight of a solution which consists of:- 10% of the diethylcyclohexylamine salts of an alkyl sulphuric acid mixture with alkyl residues C_3-C_{11}; 40% of the magnesium salts of an alkyl sulphuric acid mixture with alkyl residues C_{10}-C_{17}; 40% of water; and 10% hexalin. The edge mill treatment is continued until an even distribution of the alkylsulphuric acid salt mixture and the evaporation of the water have occurred. The red iron oxide shows no alteration of its colour properties and possesses a good distribution and floating capacity in lacquers.

Rubbings of this red iron oxide in a resin ester-linseed oil lacquer (1 : 1), rubbed in the ratio of 50 : 50, required, by the same method of testing as in Example 1, a cone loading of 21 gm., whereas an identical rubbing with untreated pigment in the same ratio required a cone loading of 150 gm.

What we claim is:—

1. A process for improving the surface properties of a powder comprising impregnating a powder with a mixture of a metal salt of a sulphonic acid or of an acid sulphuric acid ester and a salt of a sulphonic acid or of an acid sulphuric acid ester with ammonia or an organic base.

2. A process as claimed in Claim 1, in which the impregnating metal salt is an alkali-, alkaline earth- or earth-metal salt of an alkyl sulphuric acid, alkyl sulphonic acid or alkylbenzene sulphonic acid, and the impregnating ammonium or organic-base salt is a salt of an alkyl sulphuric acid, alkyl sulphonic acid or alkylbenzene sulphonic acid.

3. A process as claimed in either of Claims 1 or 2, in which the mixture of said salts impregnates the powder in quantities of 0.05% to 10%, calculated on the weight of the dry powder.

4. A process as claimed in Claim 3, in which the mixture of salts impregnates the powder in quantities of from 0.1% to 5%, calculated on the weight of the dry powder.

5. A process as claimed in either of Claims 1 or 2, in which a solution of the mixture of said salts in water or in organic solvents is mixed with the powder.

6. A process as claimed in any of the preceding claims in which the powder is a powdered colour pigment for lacquers, lacquer colours, oil colours, or synthetic resin lacquer colours.

7. A process as claimed in any of the preceding claims in which the powder is a filler for natural or synthetic rubber or rubber substitutes.

8. A process for improving the surface properties of a powder substantially as hereinbefore described.

9. Powders impregnated according to the process of any one of Claims 1-8.

W. P. THOMPSON & CO.,
12, Church Street, Liverpool, 1,
Chartered Patent Agents.

Printed for Her Majesty's Stationery Office by Wickes & Andrews, Ltd., E.C.4. 684/2.—1957.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.